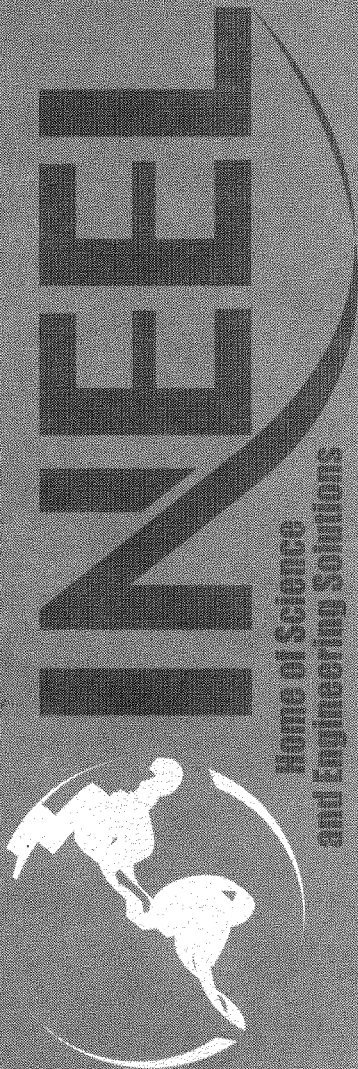


***Test Plan for the Study
of OU 7-13/14 Soil and Waste
Material Samples Obtained
from the OU 7-10 Glovebox
Excavator Method Project***

*Gary S. Groenewold
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September 2003*



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Assistant Secretary for Environmental Management
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ABSTRACT

This test plan describes experiments to measure the metal content of 36 interstitial soil and six waste samples collected during the Operable Unit 7-10 Glovebox Excavator Method Project excavation at Operable Unit 7-10 within the Subsurface Disposal Area at the Idaho National Engineering and Environmental Laboratory. The test plan includes tentative plans beyond the accelerated and enforceable schedules for Operable Unit 7-13/14 that may or may not be funded.

Particular emphasis will be placed on actinide contaminants. Subsequent leachability testing will establish how contaminant metals partition between the solid matrix and soil water over a range of pH- and ionic-strength conditions. Operationally defined speciation will be determined through performance of sequential aqueous extractions, which will enable correlation with observed leaching behavior and allow prediction of changes in partitioning that might occur because of system perturbations. The influence of colloids and organoactinide complexes (i.e., molecules composed of an actinide and one or more organic ligand) in fast transport of small fractions of actinides will be evaluated. The outcome of these studies will be:

- Characterization of the actinide content in waste and soil samples
- Evaluation of release of metal from waste solids as a primary source
- Evaluation of release of metal from interstitial soil as a secondary source
- Improvement in understanding transuranic radionuclide solubilization resulting from in situ treatment.

These data will be used to evaluate uncertainties associated with contaminant migration and to support remedial decisions.

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ACRONYMS

ASTM	American Society for Testing and Materials
EDTA	ethylene diaminetetraacetic acid
ESI-MS	electrospray ionization mass spectrometry
EPA	U.S. Environmental Protection Agency
FSP	field sampling plan
IAG	interface agreement
ICP-MS	inductively coupled plasma-mass spectrometry
INEEL	Idaho National Engineering and Environmental Laboratory
INTEC	Idaho Nuclear Technology and Engineering Center
IRC	Idaho National Engineering and Environmental Laboratory Research Center
MCP	management control procedure
OU	operable unit
QC	quality control
RFP	Rocky Flats Plant
RWMC	Radioactive Waste Management Complex
RWSC	radioactive waste and soil characterization
SAE	sequential aqueous extractions
SAP	sampling and analysis plan
SDA	Subsurface Disposal Area
SIMS	secondary ion mass spectrometry
TRA	Test Reactor Area
TRU	transuranic

Test Plan for the Study of OU 7-13/14 Soil and Waste Material Samples Obtained from the OU 7-10 Glovebox Excavator Method Project

1. INTRODUCTION

This document provides the test plan for studying Operable Unit (OU) 7-13/14 interstitial soil and waste material samples obtained by the OU 7-10 Glovebox Excavator Method Project from Pit 9 within the Subsurface Disposal Area (SDA) of the Radioactive Waste Management Complex (RWMC) at the Idaho National Engineering and Environmental Laboratory (INEEL). This test plan presents a phased approach that includes tentative plans beyond the accelerated and enforceable schedules for OU 7-13/14 that may or may not be funded.

The RWMC was established in the early 1950s as a disposal site for radioactive waste generated by operations at the INEEL and other U.S. Department of Energy laboratories. Figure 1 contains a map of the INEEL that shows the location of the RWMC and major INEEL facilities. Radioactive waste materials were buried in underground pits, trenches, soil vault rows, and one aboveground pad (Pad A) at the SDA, and transuranic (TRU) waste is now kept in interim storage in containers on asphalt pads at the Transuranic Storage Area within the SDA.

Ultimate disposition of actinide metals buried at the RWMC is currently uncertain. One possible outcome is disinterment, characterization, and shipment to a TRU-waste disposal facility (e.g., Waste Isolation Pilot Plant). A second option is to employ a form of in situ stabilization. Evaluation of uncertainties associated with selecting remedial action to minimize risk derived from the waste would be greatly improved by additional information describing the nature and extent of TRU contamination and the leaching behavior and speciation of the actinide metals of concern.

Interstitial soil is a point of emphasis for this project because soil is adjacent to and in contact with the buried waste. In the context of the RWMC, “interstitial” refers to soil between waste containers (e.g., drums and boxes). If metals are strongly retained by interstitial soil, this would suggest that interstitial soil is an effective containment barrier capable of attenuating movement of TRU metals released from the source. If this does indeed occur, and the interstitial soil is contaminated, then an explicit understanding would be needed about the release of TRU contaminants from what would then constitute a secondary source. In addition, long-term prediction of TRU solubilization must be based on the behavior of specific chemical species in which TRU metals exist.

Contaminant identity, concentration, leachability, and speciation for the waste forms are also of acute interest, and this information must be correlated with the nature of contamination to be determined from examination of the interstitial soil. Two general categories of waste are expected to be prevalent in material retrieved from Pit 9 by the OU 7-10 Glovebox Excavator Method Project, and three samples of each type will be collected for characterization and leach testing.

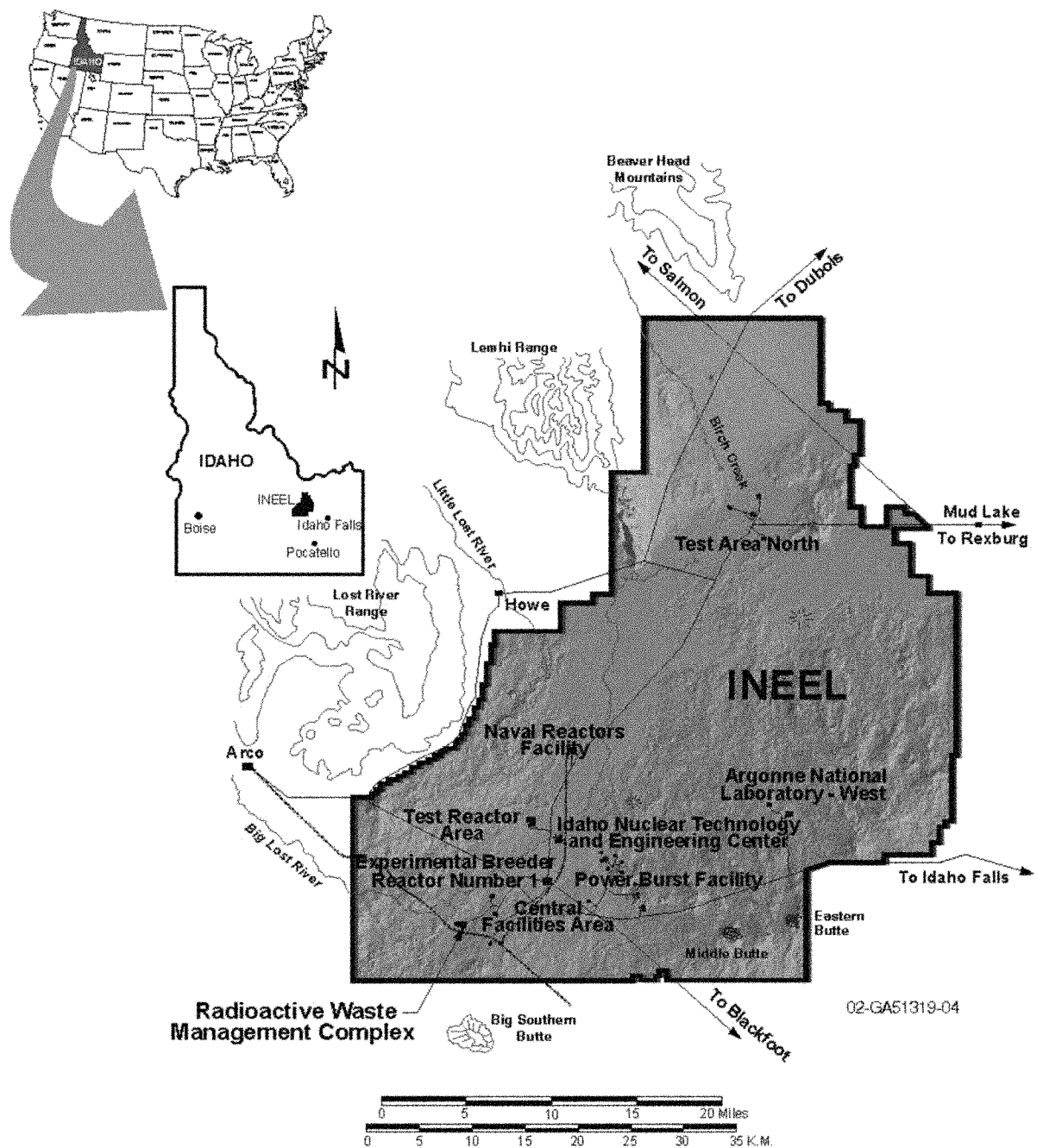


Figure 1. Map of the Idaho National Engineering and Environmental Laboratory showing locations of the Radioactive Waste Management Complex and other major facilities.

2. OBJECTIVES

The objectives of the tasks outlined in this test plan are to (1) identify constituents and concentrations of actinide radioisotopes (e.g., uranium, neptunium, plutonium, and americium) in materials retrieved from Pit 9, and (2) acquire data for evaluating contaminant migration from both types of waste material and associated interstitial soil

Data-generating tasks and associated objectives and data uses are correlated below in Table 1. Data acquired under this test plan will provide the following:

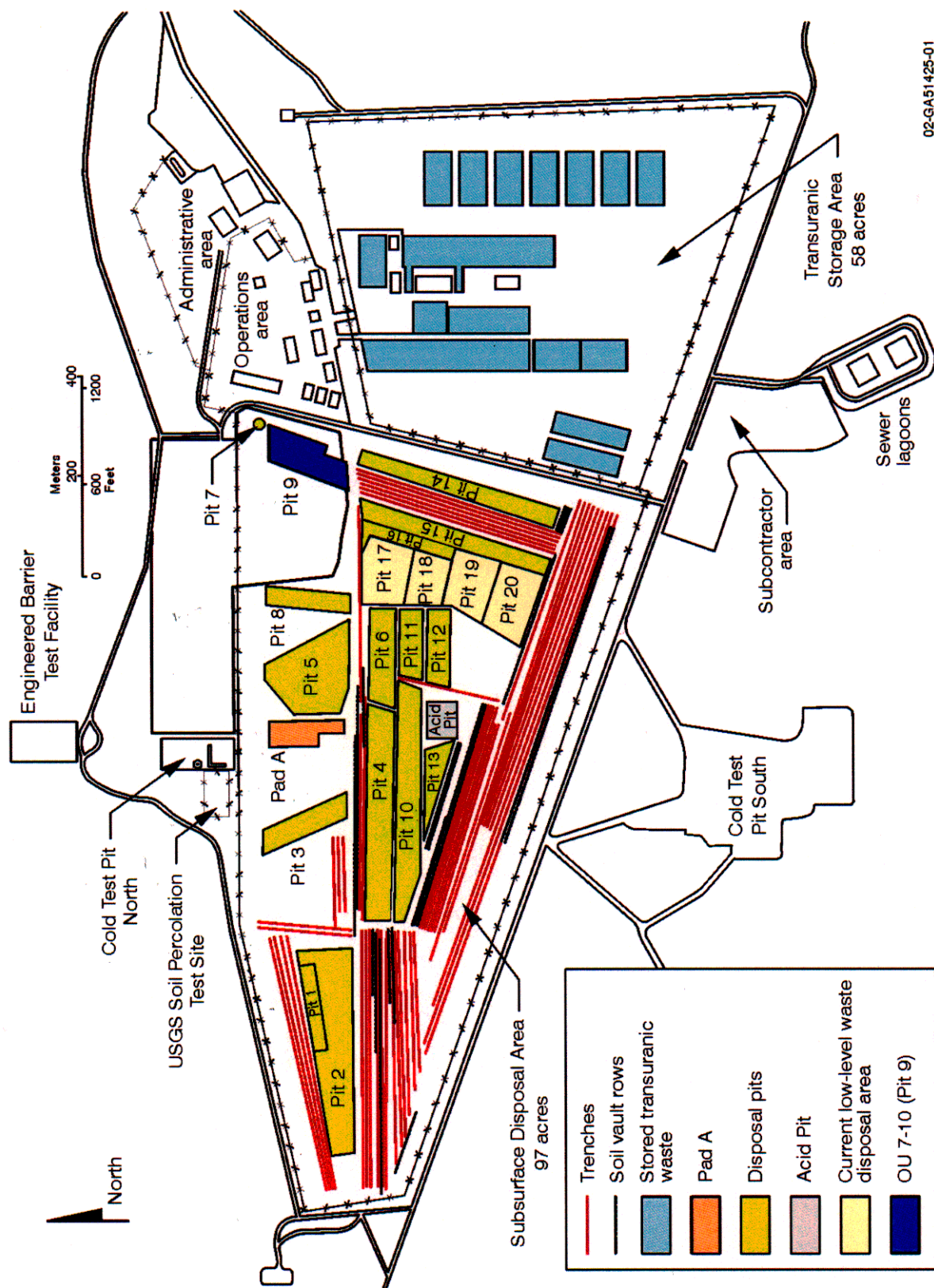
1. Total concentrations of TRU elements in sampled interstitial soil and waste: These data provide the basis for determining actinide speciation and mobility relative to waste materials and soil. This information is needed on a sample-specific basis because sample-to-sample homogeneity in material originating from OU 7-10 cannot be assumed.
2. Information about conditions under which TRU elements will be released from interstitial soil and waste: Transuranic elements will be leached from soil and waste using aqueous solutions representing a range of geochemical conditions that might be created under remediation scenarios including high pH, low pH, reducing conditions, oxidizing conditions, and high salt content. These data are used to provide design criteria for remedial alternatives because invasive alternatives may affect the mobility of TRU elements by altering the geochemical environment. These data also will provide quantitative release parameters for a more detailed source model.
3. Evaluations of the influence of colloids and organoactinide complexes (i.e., molecules composed of an actinide and one or more organic ligand) in mobilizing small fractions of TRU metal contaminants: These data are needed because fast-moving fractions of actinides can lead to the conclusion that the behavior of the bulk of the contamination is not understood. Development of a more comprehensive model, which would account for both the immobile bulk and a low-abundance, fast-moving fraction, would be enabled.

2.1 Description of Soil and Waste Samples

This test plan describes characterization of interstitial soil and waste solids to be collected from Pit 9 during the OU 7-10 Glovebox Excavator Method Project excavation. Operable Unit 7-10, which comprises Pit 9, is located in the northeast corner of the SDA (see Figure 2). The SDA has been used for shallow burial of radioactive waste from INEEL generators and other U.S. Department of Energy sites. Most waste disposed of at the SDA was originally packaged in drums or cardboard boxes and dumped or stacked in pits and trenches. The waste then was covered with native soil at the end of each operating week. No activity-based waste acceptance criteria existed at the SDA until 1957, and items with activities of up to 12,000 R/hour were reportedly disposed of at the SDA.

Table 1. Summary of data-generating tasks, objectives, data uses, and experimental approaches for evaluation of interstitial soil and waste samples derived from the OU 7-10 Glovebox Excavator Method Project.

Task	Objectives	Data Uses	Test Method
Determine total actinide concentration	These data are necessary to identify actinides present in different media and to determine their concentrations.	These data will provide the baseline information on actinide concentration critical for mass balance estimation during actinide speciation and mobility relative to waste materials and soil.	Total actinide content will be measured in all waste media and soil. Subsamples will be completely dissolved and the resulting solution analyzed using multi-element ICP–MS.
Characterization of sample mineralogy, surface chemistry, and selected chemical and physical properties	These data are necessary to evaluate conditions under which TRU elements will be released from interstitial soil and waste media.	These data are important in understanding the actinide-release behavior for assessing risk, evaluating remediation options, and assessing potential unintended consequences resulting from treatment options that might result in alterations in the pH, Eh, transition metal, or organic content of the waste-bearing region.	Leach test under remediation scenarios including high pH, low pH, reducing conditions, oxidizing conditions, and high salt content. Soil mineralogy will be determined by conventional x-ray diffraction techniques. Surface chemistry will be evaluated using SIMS.
Tentatively planned column studies	This task will evaluate mobility of actinides.	These experiments will use a variety of actinide species to evaluate mobility of actinides.	Analyses will use ICP-MS.
Tentatively planned evaluations of the influence of colloids and organoactinide complexes in mobilizing small fractions of TRU metal contaminants	This task will determine whether organic ligands or submicron particles are causing fast transport for a small fraction of the actinide metals.	This task will determine whether organic ligands or submicron particles are causing fast transport for a small fraction of the actinide metals. These data are needed to support both the risk assessment and feasibility study.	Colloids will be suspended in low ionic-strength water followed by separation using either filtration or centrifugation. Colloids will be dissolved and analyzed using ICP–MS for actinide content. In addition, colloids will be analyzed using microscopy, surface analysis (i.e., SIMS), x-ray diffraction, and in situ laser scattering. Organoactinide complexes will be separated using selective extraction and chromatographic techniques. Complexes will be analyzed using electrospray ionization mass spectrometry.
ICP-MS = inductively coupled plasma-mass spectrometry SIMS = secondary ion mass spectrometry TRU = transuranic			



02-GA51425-01

Figure 2. Graphic of the Radioactive Waste Management Complex showing the Subsurface Disposal Area.

Transuranic waste from Rocky Flats Plant (RFP)^a also was disposed of at the SDA. The RFP waste consisted of three types of sludge (i.e., inorganic, organic, and nitrate salts) together with debris-type waste. The sludge and debris (e.g., cloth, metal, wood, asphalt, glass, and plastic) were containerized in metal drums or wooden crates and stacked horizontally in pits and trenches among the waste from the INEEL.

Two types of sludge are of particular interest because they have a good probability of containing plutonium and other TRU contaminants. To simplify the discussion, these have been termed organic sludge and cemented sludge (Arrenholz and Knight 1991). The chemical form of TRU elements is likely to be different in the two types of sludge. Organic sludge would mainly contain oxidized plutonium metal in the form of PuO₂, while the inorganic sludge would contain TRU oxyhydroxides precipitated from aqueous solution. Organic sludge is derived from Series 743^b organic setups and consisted of chlorinated solvent waste solidified using calcium silicate. Quite a few drums of Series 743 sludge are buried Pit 9, and it is estimated that from 50 to 80 drums of Series 743 sludge will be excavated (Salomon et al. 2003). The cemented sludge was derived from either the Series 741 first-stage sludge or the Series 742 second-stage sludge generated from salt precipitates containing plutonium oxides, americium oxides, metal oxides, and possibly mixed with depleted uranium. These materials were solidified using Portland cement to soak up and solidify residual liquid. Only a few drums of Series 741 and 742 sludge are in the excavation area (Salomon et al. 2003); therefore, excavation of a drum of Series 741 or 742 sludge would be of particular interest.

In addition to the waste materials, interstitial soil will be exhumed. Although several different soil lenses are found in native surficial sediments at the RWMC, the manner in which the pits and trenches were dug and then backfilled over the waste caused some mixing of various surficial soil strata. The typical RWMC surficial sediment is predominantly quartz-like (see Table 2), but sizeable clay and carbonate fractions can significantly influence metal-adsorption chemistry (Groenewold and Redden 2002; Rightmire and Lewis 1987). Mobilized TRU metals possibly have infiltrated interstitial soil where readsorption or mineral precipitation has occurred. This action, if it has occurred, would have produced a secondary source that will have to be characterized to fully understand the risk derived from it. The extent of contamination and the leachability of TRU metals from the interstitial soil samples constitute a major emphasis of the task detailed in this plan. It is emphasized that sample-specific measurements are needed for accurate release measurements and for assessment of sample heterogeneity.

2.2 Contaminants of Interest in the Radioactive Waste Management Complex Samples

The *Ancillary Basis for Risk Analysis of the Subsurface Disposal Area* (Holdren et al. 2002) identified human health and ecological contaminants of concern associated with buried waste. Contaminants of concern addressed by this test plan are the actinides listed in Table 3 (i.e., isotopes of uranium, neptunium, plutonium, and americium). Leachability testing and solid-phase association can be correlated with the chemical form and solubilization of the actinide elements. Therefore, information will be generated on leaching and speciation of isotopes of concern in the RWMC samples.

a. The Rocky Flats Plant is located 26 km (16 mi) northwest of Denver. In the mid-1990s, it was renamed the Rocky Flats Environmental Technology Site. In the late 1990s, it was again renamed, to its present name, the Rocky Flats Plant Closure Project.

b. The waste is called Series 743 waste because it was processed into sludge in RFP Building 774 and was later coded at the INEEL as Content Code 3 organic waste to distinguish it from different types of waste from RFP Building 774 shipped to the INEEL.

Table 2. Description of surficial soil material typical of the Subsurface Disposal Area.

Property	Description
	Silty loam
Texture particle size distribution	Sand ($>50\ \mu\text{m}$) = 26% Silt ($50 - 2\ \mu\text{m}$) = 60% Clay ($<2\ \mu\text{m}$) = 15%
pH	7.2 to 8.2
Cation exchange capacity	7 – 14 meq/100g
Exchangeable cations	$\text{Ca}^{2+} \gg \text{Mg}^{2+} > \text{Na}^+ > \text{K}^+$
Carbonates	0.1 to 5.7 wt%
Organic matter	0.3 to 1.5 wt%
Total soil iron	3.8 to 4.0 wt%, mostly as oxide
Mineralogy	$>75\%$ quartz, 8% plagioclase, 3% orthoclase, 1% mica, with balance accounted for by clay and calcite
Clay mineralogy	30% illite, 40% mixed-layer illite/smectite, 10% calcium and sodium smectite, 20% kaolinite

Table 3. Actinide isotopes of concern in the Subsurface Disposal Area samples.

Actinide Element	Isotopes of Concern
Uranium	234, 235, 236, and 238
Neptunium	237
Plutonium	238, 239, and 240
Americium	241

3. SCOPE

Project activities cover characterization of samples acquired from Pit 9 by the OU 7-10 Glovebox Excavator Method Project. The subtask structure provides the following broad categories of sample information:

1. Total actinide concentrations in each sample
2. Quantities of actinides released from the samples under a range of pH- and ionic-strength conditions
3. Identity of interstitial soil and waste phases associated with actinides
4. Description of geochemical properties of interstitial soils
5. Formation and mobility of colloidal forms of actinides
6. Formation of mobile aqueous complexes between actinides and organic molecules.

A staged approach will be used to provide a comprehensive understanding of actinide content, concentration, and speciation for the collected samples.

Note: In the context of this test plan, speciation refers to the envelope of oxides, oxidation states, mineral forms, ligand complexes, and colloidal forms that the metal can assume. Knowledge of speciation is critical to forecasting release under changing conditions and perturbations (e.g., various remediation scenarios), which is an essential element of this work.

Knowing the total actinide concentrations in each sample is vital sample-specific information needed to measure the extent of actinide release. Knowing the actinide-release behavior is important for assessing risk, evaluating remediation options, and assessing potential unintended consequences resulting from treatment options that might result in alterations in the pH, Eh, transition metal, or organic content of the waste-bearing region.

Phenomenological mobility behavior across a range of pH- and ionic-strength dissolution conditions will be determined. This will produce a range of partition coefficients (i.e., K_d values) at relevant pH-, Eh-, and ionic-strength conditions representative of the excavated area of OU 7-10. An evaluation of actinide mobility from the samples also will be conducted using both batch and column experiments.

Small fractions of the actinide contamination may display mobility that is substantially faster than average. Soluble carbonate species are known to form in calcareous soil such as that found at the INEEL. Colloids and organoactinide complexes also have been widely implicated as potential transport vectors for small fractions of actinide contamination. If fast-moving actinide fractions are detected from the column experiments mentioned above, then a subtask would be undertaken that involves rigorous identification of the chemical nature of the minor fraction and assessment of its mobility in response to potential geochemical alterations.

Subtasks comprising the scope of this test plan are described in the following subsections.

3.1 Sample Acquisition by the OU 7-10 Glovebox Excavator Method Project

Although considerable process knowledge and analytical data related to the soil and waste solids (see Section 2.1) do exist, those data are insufficient to realistically describe migration of isotopes of interest from that waste and soil. Additionally, no existing data describe migration of isotopes of interest under the variety of geochemical perturbation conditions that may arise from in situ treatment. Thus, acquisition of waste solids and interstitial soil samples from Pit 9 by the OU 7-10 Glovebox Excavator Method Project affords a unique opportunity to acquire data that currently do not exist yet are useful for understanding past and forecasting future migration.

For activities described in this test plan, the purpose of analyzing material samples from Pit 9 is to characterize and assess specific traits possessed by those samples. Those specific traits are actinide identity, speciation, concentration, and subsequent mobility under a given set of geochemical conditions. Data generated from samples are to be used for evaluating contaminant migration, whether the materials remain in the ground untouched or undergo some future perturbation introduced by in situ treatment.

The *OU 7-10 Glovebox Excavator Method Project Conceptual Design Report* (INEEL 2002), states that approximately 75 to 125 yd³ of waste zone material will be retrieved (e.g., approximately 230 drums of waste zone material and approximately 30 yd³ of interstitial soil). Of the 230 drums, it is expected that two will contain Series 741 and 742 sludge, and that 50 to 80 drums will contain Series 743 sludge. For retrieval activities, the excavator will be equipped with a clamshell digging bucket capable of both scooping soil and debris and grabbing intact objects as large as a 55-gal drum. Waste zone materials will be loaded into a 30 × 40 × 30-in. waste-transfer cart, which then will be transported to the Packaging Glovebox System where materials will be opened, inspected, sorted, sampled, sized, monitored, and repackaged. Inside the glovebox, intact and deteriorated 55-gal drums will be opened and the contents removed for inspection and sorting. Soil and other waste-zone materials also will be received in the glovebox and inspected, sorted, sampled, and repackaged.

Conceptually, acquisition of Series 741/742 and 743 sludge samples will be relatively straightforward and simple if intact or partially intact drums of those materials are exhumed. Regardless of the location in the pit from which material is excavated, it is important that sludge drums and interstitial soil immediately adjacent to those drums are acquired. Because multiple waste forms may be encountered, there may be substantial variability in the interstitial soil, which indicates the need to collect sufficient samples to assess soil heterogeneity.

Sampling and related quality control (QC) activities are defined by the *OU 7-10 Project Field Sampling Plan* (FSP) (Salomon et al. 2003) and are beyond the scope of this test plan. It should be noted that the location and frequency of sampling waste zone materials is given in the FSP, Section 3.2, “Waste Zone Material Sampling.” The FSP indicates that the OU 7-10 Project will acquire samples of interstitial soil and waste solids to satisfy the data quality objectives stated in Appendix B of that document. A total of 42 (containing approximately 250 cc each) waste solids and interstitial soil samples from the project will be collected and used in activities described in this test plan in partial satisfaction of the overall project data quality objectives. Of these samples, three different samples of Series 741/742 and 743 sludge will be acquired, for a total of six different samples of waste solids. For each sludge sample, six different soil samples will be acquired from contaminated interstitial soil adjacent to the drums containing waste solids, for a total of 36 interstitial soil samples. The sample-selection scheme is purposely biased for sludge because it is estimated that it will constitute the highest overall weight percentage of the waste solids excavated from the pit (Salomon et al. 2003) and is expected to contain sufficient concentrations of the

various isotopes of interest. The sample selection scheme is also purposely biased for the interstitial soil because of the need to acquire contaminated soil (vs. uncontaminated soil) for actinide-mobility studies.

Acquisition of 36 contaminated interstitial soil samples in groups of six, each group associated with an identifiable drum of a particular sludge, will provide data to examine the difference in mobility of actinides based on sludge type. From this information, sludge-specific source-release parameters could be derived for inorganic (Series 741 and 742) and organic (Series 743) sludges. Even if samples are not linked to a specific drum of sludge, if a link can be made to a specific sludge type, then it may still be possible to develop sludge-specific source term release parameters. If contaminated interstitial soil samples cannot be tied to a specific sludge or waste type, then it will only be possible to develop generic source-term-release parameters. This will still be valuable. Clean interstitial soil will not provide material to develop an understanding of contaminants in interstitial soil or allow development of source-release parameters. Therefore, samples are necessarily biased toward areas of likely contamination (e.g., stained soil immediately adjacent to breached drums). However, samples that contain waste and soil intermixed, while probably being contaminated, will not provide material to measure release parameters because the waste will mask the release of contaminants from the soil.

3.2 Sample Shipment to the Test Reactor Area and Return of Excess Material

As tentatively planned, three samples each of Series 741/742 and 743 sludge and 36 contaminated interstitial soil samples from the project excavation, acquired specifically for this task, will be prepared and handled as described in the *OU 7-10 Glovebox Excavator Method Project Conceptual Design Report* (INEEL 2002) and the OU 7-10 FSP. The samples will be shipped directly to CPP-602, as described in Interface Agreement (IAG) -149, "Interface Agreement, Sample Handling and Characterization Agreement Between the OU 7-10 Glovebox Excavator Method Project and the Analytical Laboratories Department." After the samples have been received at CPP-602, the samples designated for this activity will be identified and subjected to a gamma spectroscopy scan. Results will be used to determine whether samples can be shipped to the Test Reactor Area (TRA), because those results will provide an estimate of the amount of plutonium likely to be present in the sample. Acceptable samples then will be shipped to the TRA α wing without further characterization or manipulation by the INEEL Analytical Laboratories Department. Samples will be shipped from CPP-602 to the α wing of TRA in accordance with all applicable guidelines for shipment of radiological material within INEEL boundaries. Once at TRA, the samples will be subsampled and characterized as described in Section 3.3. Any unused material will be returned directly to OU 7-10 from either CPP-602 or from TRA. Waste materials generated in this project will be handled as described in the waste management plan (Hanson et al. 2003).

3.3 Visual Inspection and Subsampling at the Test Reactor Area

As tentatively planned, all samples received at the TRA α wing from CPP-602 will be visually inspected for the presence of graphite mold fragments. If graphite mold fragments are found, Security will be contacted to clear the samples. Graphite mold pieces, if released by Security, will be segregated from the sludge or interstitial soil and will comprise a new sample subclass. Graphite mold pieces are suspected of containing plutonium, for which the release behavior will be considerably different than the release behavior from either soil or sludge solids. After visual inspection and segregation of graphite, and individual sludge and soil samples, three subsamples will be collected from each sample. Subsamples will be used for characterization as described in Section 3.4.

Subsampling, graphite segregation, and sample handling will be performed in a suitable hood or glovebox where radioactive materials can be handled safely and radiological contamination can be controlled.

3.4 Analysis for Total Actinide Content in Subsamples

As tentatively planned, total actinide content will be measured in all 42 of the 250-cc sludge and soil samples and on the graphite mold fragments (if any). Subsamples will be completely dissolved and the resulting solution analyzed using sensitive multi-element inductively coupled plasma-mass spectrometry (ICP-MS) detection. This first round of subsampling and analysis will provide an indication of the actinides present in each sample, their concentration, and heterogeneity. The statistical mean concentration of actinides and standard deviation will be determined through analysis of three replicate subsamples of each sample. These data also will be used to determine whether or not the degree of heterogeneity in any given sample is high enough to warrant further subsampling and analysis. Total actinide content will be measured in the graphite mold fragments by selecting at least three different pieces, weighing the pieces, and then digesting (i.e., dissolving) and analyzing the pieces using ICP-MS.

Total actinide data are the basis for obtaining mass balance for actinide-migration studies. The actinide content and concentration associated with waste, soil, or graphite both before and after leaching is vital information that allows computation of distribution coefficients. Determining actinide content and concentration also allows for identification of different species present in the samples before exposure to leaching solutions, and subsequent speciation changes promoted by exposure to leaching solutions.

3.5 Characterization of Sample Mineralogy, Surface Chemistry, and Selected Chemical and Physical Properties

Actinide release from mineral samples is a strong function of the mineralogy and surface chemistry of the matrix. Development of predictive tools, used for modeling contaminant transport and assessing the impact of perturbations to the system, will rely on understanding the chemical and physical properties of the samples as well as sample mineralogy and surface chemical characteristics.

Several different soil lenses are known to exist in native surficial sediments at the RWMC. Differences in soil texture and mineralogy can be found from the surface down to the underlying basalt layer. Soil lenses range from having a high percentage of clay to having a low percentage of clay with a high percentage of calcium carbonate. The manner in which the pits and trenches were dug and then backfilled over the waste caused partial mixing of various surficial soil strata. Thus, the backfill material may resemble a composite sample of all SDA surficial sediments, but could also contain sizeable clay and carbonate fractions capable of significantly influencing metal-adsorption chemistry. Because soil mineralogy, surface chemistry, and chemical and physical properties can significantly influence metal transport, those properties will need to be determined for a selected number of interstitial soil samples acquired from the project excavation.

As tentatively planned, the mineralogy, surface chemistry, and selected chemical and physical properties of selected samples will be assessed using applicable techniques from the American Society for Testing and Materials, U.S. Environmental Protection Agency (EPA), and surface spectroscopy. Sample selection criteria for the tests will be based on the concentration of actinides found in each of the 36 interstitial soil samples. Tests will include determination of soil pH, cation-exchange capacity, specific surface area, soluble cations and anions, weight-percent soil carbonate, weight-percent soil organic matter, weight-percent soil iron, soil mineralogy, and soil-surface chemistry. Soil mineralogy will be determined by conventional X-ray diffraction techniques, and surface chemistry will be evaluated using secondary ion

mass spectrometry (SIMS) at TRA. Resources permitting, Mossbauer spectroscopy will be performed on a limited subset of the samples.

Data pertaining to soil mineralogy and other selected physical and chemical properties of the soil will be used to develop predictive actinide-release models. Surface chemistry also will contribute through identification of the envelope of surface cations present that would compete with actinides in exchange processes.

3.6 Contaminant Leachability Across pH- and Ionic-Strength Gradients

Transport of radionuclides can be significantly enhanced with only minor changes in the soil chemical environment. Changes in soil pH, Eh, and salinity can be brought about by many different factors, whether they are anthropic or natural (e.g., cyclic wetting and drying, intrusion of chemical agents, or microbial action). Two specific factors that have a high probability for perturbing the soil environment and potentially causing significant changes in actinide mobility are changes in soil pH and pore-water ionic strength. To accurately predict the magnitude and extent to which those factors influence metal transport, information is needed that describes partitioning behavior under conceivable perturbation conditions. Partitioning of actinides to surficial sediment is measured as a function of (1) changes in the pH and (2) ionic strength of synthetic groundwater.

As tentatively planned, the interstitial soil samples and six waste samples will be subjected to simulated groundwater leaching across a range of pH values from 10 to 3. Leachability tests as a function of pH will use aqueous solutions of simulated groundwater modified with NaOH to pH 10. The pH will be adjusted downward to pH 3 in one-pH-unit steps using HCl. Metal content in the leaching solution and metal partitioned to the solid will be analyzed at each step using ICP-MS. Up to six samples identified as having significant actinide content also will be subjected to a simulated groundwater leach across a range of ionic-strength values ranging from solution conductivities from approximately 10 to 5,750 μS . Leaching will initially use low ionic-strength water and then will increase ionic strength by factors of 3 \times , 5 \times , 10 \times , and 20 \times that of simulated groundwater.

Solid to solution-partitioning data (K_d values) are needed to evaluate potential for the release of actinides from soil and waste matrices and to understand the sensitivity of partitioning to changes in pH and ionic strength. The quantity of actinides solubilized at the lowest pH- and highest ionic-strength value is likely to represent the maximum amount released under conceivable conditions. This will enable data describing the nonleachable actinide contamination to be treated in a less conservative fashion because it is unlikely to be solubilized.

3.7 Sequential Aqueous Extractions

As tentatively planned sequential aqueous extractions will be performed on all 36 of the interstitial soil samples and six waste samples. The purpose of conducting sequential extractions is to generate operationally defined speciation for interstitial soil and the two waste forms. This will enable differentiation of immobile actinide fractions from those liable to be mobile. Fractions of actinides in operationally defined categories will enable projection of release of actinides under different remediation options that might be oxidizing or reducing. In addition, the data will be used to reduce conservatism by putting bounds on the total amount of actinides that might be mobilized because the fraction in the more tightly bound categories is unlikely to be solubilized under any conditions and hence can be assigned a large K_d value.

Serial extractions will use (1) a cation exchange leachant (2) a carbonate dissolving leachant, (3) an oxidizing leachant, or (4) a reducing leachant. Actinide metals will be analyzed by ICP–MS in each of the leachates. Residual solids will be dissolved and analyzed to generate a total mass balance for comparison with results of the total actinide content determinations.

3.8 Column Studies

Batch solubilization studies provide information on the extent of actinide release, but do not provide direct information about the mobility of the actinides through the RWMC sediments. An approximation of mobility can be generated using experiments that measure actinide transport through columns of sediments. Hence, column experiments are tentatively planned consisting of typical OU 7-10 sediments will be conducted to evaluate mobility of actinides. These experiments will use a variety of actinide species as subjects for mobility investigations. Included will be actinides solubilized from the OU 7-10 samples, which may include solution-phase and or colloidal forms.

3.9 Colloids and Organic Complexes

This tentatively planned task will determine whether or not organic ligands or submicron particles are causing fast transport for a small fraction of the actinide metals. These factors have the potential to be critically important because if they mobilize even a trace quantity of actinide contamination, that would suggest a significant plume might be mobilized. Specific samples to be analyzed for colloids and organoactinide complexes will be determined by results from the total actinide content, sequential aqueous extractions, and column-leaching experiments. Samples with substantial actinide contamination and substantial oxidizable fraction will be examined for colloids and complexes, respectively.

As tentatively planned, colloids will be suspended in low ionic-strength water followed by separation using either filtration or centrifugation. Colloids will be dissolved and analyzed using ICP–MS for actinide content. In addition, analysis of colloids will be performed using microscopy, surface analysis (i.e., SIMS), X-ray diffraction, and in situ laser scattering. Separation of organoactinide complexes will be accomplished using selective extraction and chromatographic techniques. Complexes will be analyzed using electrospray ionization mass spectrometry (ESI-MS).

Mobility of actinides caused by colloidal material will be used to reduce conservatism by putting bounds on the total amount of actinides that might be mobilized by either colloids or organoactinide complexes. This use assumes that mobility of the actinides will be low, which is a good assumption because of the likely high K_d values of solution-phase actinides in the RWMC sediments.

4. ORGANIZATION, RESPONSIBILITIES, AND TRAINING

4.1 Responsibilities

The INEEL chemistry and geochemistry staff developed the studies defined in this test. Principal investigators were Gary S. Groenewold, Ph.D. (chemistry), and Larry Hull, Ph.D. (geochemistry). Multiple organizations will be used to accomplish the test objectives and laboratory facilities will be used at the Idaho Nuclear Technology and Engineering Center (INTEC), the Test Reactor Area (TRA), and to a lesser extent at the INEEL Research Center (IRC). Table 4 identifies key project staff and their primary project responsibilities.

Table 4. Key project staff and responsibilities.

Project Responsibility	Team Member
Clean/Close RWMC project director	John M. Schaffer
OU 7-13/14 project manager	Frank L. Webber
OU 7-13/14 RI/FS project manager	Brandt G. Meagher
RWSC test leads	Gary S. Groenewold, Ph.D., chemistry; Larry C. Hull, Ph.D., geochemistry
Leaching studies and sequential aqueous extractions	Robert V. Fox, Ph.D., chemistry
TRA operations	Bruce J. Mincher, Ph.D., radiochemistry
Analytical chemistry	William F. Bauer, Ph.D., chemistry
INTEC operations	Rick L. Demmer, M.S., chemistry
Waste Generator Services	Jeffrey C. Messaros, Rhonda D. Rohe
Environmental	Brent N. Burton

INTEC = Idaho Nuclear Technology and Engineering Center

OU = operable unit

RI/FS = remedial investigation/feasibility study

RWMC = Radioactive Waste Management Complex

RWSC = radioactive waste and soil characterization

TRA = Test Reactor Area

4.2 Training

Training requirements are established by each testing organization and are specific to the testing facility and identified hazards posed by testing. Before initiating testing at INEEL facilities, hazard checklists and mitigation plans will be prepared as prescribed by INEEL Management Control Procedure (MCP) -3571, "Independent Hazard Review." Specific training requirements not already addressed by the facility's authorization basis will be identified through the independent hazard review. Training records for INEEL testing personnel will be reviewed before initiation of the project to ensure that job-specific training requirements have been addressed and are up-to-date.

5. TEST DESCRIPTION

5.1 Sample Selection and Collection

Ideally, observation of the excavation process by project personnel would enable visual identification of material sampled, in the context of other items encountered in the retrieval project. For this reason, personnel will observe the sampling process as it occurs. If possible, guidance will be provided to the glovebox excavator personnel. However, sample collection and associated QC are specified by the OU 7-10 FSP and not by this test plan. The FSP also specifies sample packaging and shipment to INTEC.

All equipment, materials, and supplies needed for this task are specified in the FSP. Facility requirements are not an issue for this task.

5.2 Sample Identification, Shipment to the Test Reactor Area, and Subsampling, Storage, and Return of Excess Material

Samples will initially be shipped to CPP-602 because sample receipt must proceed on a 24-hour schedule, and sample containers must be returned to the glovebox. Personnel from INTEC will remove samples from initial shipping containers and identify the radioactive waste and soil characterization (RWSC) samples for shipment to TRA. Once received at TRA, samples will be homogenized and subsampled, and the subsamples will be used for analysis, leaching, and transport experiments. Unused samples will be returned to the OU 7-10 Glovebox Excavator Method Project.

Glovebox facilities and gamma spectroscopy at INTEC will be used. In addition, space at INTEC will be used to conduct sample identification and screening.

Glovebox facilities available at TRA will be used to perform subsampling. Containers and storage space at TRA will be identified for storage of the subsamples.

5.3 Graphite: Visual Inspection and Subsampling

Fragments of graphite plutonium molds could be present in the waste and actinide concentrations, and actinide-release behavior from this material could be vastly different than the interstitial soil or waste. Therefore, samples will be visually inspected for graphite, which will have a substantially different appearance compared to the soil and sludge samples. If black fragment material is observed, it will be separated from the sample using forceps and collected in a separate bottle. Security must clear any graphite fragments identified. Work will be performed on graphite fragments only if permitted by Security. Graphite samples will be characterized in the same manner as the soil samples (i.e., total actinide content will be measured and leachability, as a function of pH and ionic strength, will be measured) and operationally defined speciation will be determined. In addition, parallel surface analyses will be performed.

A binocular optical microscope will be needed and will be procured if one cannot be located on-Site. Sample containers for graphite subsamples will be identified and laboratory space at TRA will be used to store containers.

5.4 Total Actinide Content

Total actinide content is one of the most critical parameters because it provides a realistic estimate of concentrations in the primary and secondary source terms. This estimate is needed on a sample-specific basis to measure extent of actinide release in subsequent leaching studies. The approach for generating total

actinide information will be to aggressively dissolve soil samples and then analyze the solutions using the multi-element capability of ICP-MS in a manner similar to that described for total metals analysis in EPA publication SW-846 Methods 3020, 3050, 3051, and 6020 (EPA 1996), and EPA Method 200.8 (EPA 1995). Three 0.1-g subsamples from each sample will be weighed, then digested, and then analyzed using ICP-MS. Dissolution will use either aqua regia (HNO_3/HCl) (Agarande et al. 2001; Tonouchi et al. 2002) hydrofluoric acid, or a lithium borate fusion technique. The dissolution liquids then will be measured for total metals (emphasis on actinides) using ICP-MS (Agarande et al. 2001; Baglan et al. 2000; Hinrich and Schnetger 1999; Pawlak and Rabciga 2002; Zbiral et al. 2000) The graphite subsamples will be dissolved using a similar approach.

Digests from selected subsamples will be cross-checked using conventional alpha-, beta-, and gamma-spectroscopic analysis. The principal purpose for the cross-check will be to distinguish Am-241 from Pu-241.

Replicates of the samples will be divided into batches of 18, or six samples per batch, as listed below:

- Interstitial soil 1 to 6 batches
- Interstitial soil 7 to 12 batches
- Interstitial soil 13 to 18 batches
- Interstitial soil 19 to 24 batches
- Interstitial soil 25 to 30 batches
- Interstitial soil 34 to 36 batches
- Organic waste (Series 743) 37 to 39 batches
- Cemented waste (741/742 series) 40 to 42 batches
- Graphite subsamples (six additional) batches.

Hood space to allow aggressive acid-dissolution experiments will be identified at the TRA laboratories. The ICP-MS and alpha-spectroscopy instrumentation at TRA will be used. Laboratory-ware sufficient to analyze a batch of 18 samples (including the QC load) and mineral acids for soil and waste dissolution will be identified or procured. It is anticipated that α -spectroscopy capability at the Radiation Measurement Laboratory at TRA will be used.

5.5 Mineralogical and Surface Characterization

The mineralogical and surface chemistry of the samples will be assessed because the rates of release of actinides adsorbed to mineral surfaces are strongly influenced by these factors. Correlation of actinide release, with the identity of the adsorbing mineral and its chemical surface composition, greatly strengthens the scientific basis for release behavior. Decisions that rely on source-term behavior are scientifically defensible if the mineralogical and surface chemical nature of the source matrix is known. Without mineralogical and surface chemistry information, actinide release data are merely phenomenological. Therefore, identification of mineral form and surface chemistry is an important task.

For those samples having no, or negligible, actinide contamination (as determined in Section 5.4), samples will be shipped to the IRC, where mineralogical characterization will be conducted. Only approved samples will be sent to the IRC or to outside INEEL subcontractors. Samples will meet all applicable state and federal regulations. For planning purposes, it is assumed that six samples (18 subsamples) will be radiologically cold enough to enable transport to the IRC for analysis. X-ray diffraction will be used to deduce mineral form. Ion chromatography will be used to identify extractable anions. And surface-area analysis will be used to measure specific surface area (Adamson 1990).

All soil, waste, and graphite subsamples will be interrogated using SIMS (Metson and Perry 1990; Van Vaecck, Adriaens, and Gijbels 1999) at TRA for assessment of surface composition and correlation with mineral type. Soil and waste subsamples total 126, and an additional six graphite subsamples are assumed.

The SIMS instrument at TRA will be used. At IRC, the X-ray diffraction, surface-area analyzer, and ion-chromatography instrumentation will be used. Miscellaneous consumable supplies for sample-handling and -mounting hardware will be identified or procured. Laboratory space for sample preparation at TRA and IRC will be identified.

5.6 Leachability Across pH- and Ionic-Strength Gradients

Actinide release is a critical parameter in assessing risk and remedial alternatives. Release can vary greatly across a relatively narrow range of pH values that can be easily achieved in surficial soil environments. Generally, at high pH values (e.g., pH higher than 8), release will be low or negligible. However, as pH decreases, adsorbed actinides are displaced and can then be transported. Also, as pH decreases, changes in the oxidation state can occur as well as changes in the number and type of ligands coordinated to the metal. These changes can promote higher actinide solubility and lead to transport. At lower pH values, carbonate-mineral phases dissolve and release carbonate-bound actinides, and other pH-promoted changes occur that affect the physical- and chemical-complexing behavior of the minerals and clays present in the soil—again leading to transport. The influence of pH becomes even more acute when remediation alternatives are considered that might alter the native soil pH. For example, grouting may raise the pH, which is generally good, unless soluble carbonate complexes are formed leading to higher transport rather than the desired sequestration effect.

Chemical equilibria are markedly influenced by the presence of ionic species in solution. As the concentration of ions in solution increases, chemical potentials and activity coefficients dramatically depart from unity, which noticeably alters the solubility of metals. It is well known that as the salinity of soil water increases, metal mobility increases proportionately. Thus, the mobility of actinides is greatly impacted not only by pH, but also by the presence of competing ions dissolved in the soil water. At the RWMC, where TRU metals were interred along with large quantities of inorganic salts, the concentration of ions in the soil water contacting the source term could be relatively high. To more accurately predict transport, actinide-release rates must be assessed not only as a function of pH, but also as a function of the salinity of the aqueous phase coming into contact with the waste or soil.

Three 1-g replicates from each of the 42 soil and waste samples will be subjected to a variable pH leaching experiment to determine how the actinide partitioning responds to variations in the pH and ionic strength of the soil water. This experiment will produce K_d values across the range of pH values relevant to OU 7-10. Actinide partitioning can change significantly with minor alterations in pH. These thresholds could conceivably be crossed during the course of in situ remediation activities, which would result in an unplanned migration of actinides. Therefore the K_d values, and their variability with changes in pH will be used to estimate actinide release. Actinides could be released from soil materials left behind in the event the waste is dug up, or could be unintentionally released as a result of chemical changes during an in situ treatment.

A selected set of samples will be subjected to leaching under combined, variable pH- and ionic-strength conditions. Ionic strength will be adjusted by increasing the concentration of the simulated groundwater constituents. This may result in precipitation of calcite, and so the water will be filtered to remove precipitate before initiating leaching experiments. This experiment will produce K_d values across the range of pH- and ionic-strength values relevant to OU 7-10. Because the addition of ionic strength as a variable significantly expands the number of analyses, a selected set (assumed to be six) of the samples having high total actinide content will be used for this test.

Leaching solutions will be based on simulated groundwater, which will be prepared using the average concentration of inorganic constituents found in water obtained from sampling wells in and around the RWMC (see Tables 5 and 6). A calculation will be performed to evaluate the possibility of precipitating calcite resulting in elevated pCO_2 , and the system will be carbonate buffered as appropriate to control this variable.

Table 5. Subsurface Disposal Area groundwater simulant recipe for in situ grouting and in situ thermal desorption leach testing.

Salt	Grams per 50 L Water	Salt	Grams per 50 L Water
$MgSO_4 \cdot 7H_2O$	11.252	$NaHCO_3$	4.62
$CaCl_2$	9.7	$KHCO_3$	0.31
$NaNO_3$	0.17	KNO_3	0.20
Nanopure H_2O	50 L	—	—

Table 6. Simulated groundwater characteristics.

Water Characteristics	Concentration (ppm)	Water Characteristics	Concentration (ppm)
SO_4	88	HCO_3	71
Cl	124	K	4
NO_3	5	Ca	70
Na	26	Mg	22
Total dissolved solids	410	—	—
Conductivity	567 $\mu S/cm$	—	—
pH	7.55 – log	—	—

Graphite subsamples also will be subjected to the variable pH- and ionic-strength leach, if sufficient sample is available.

Replicates of the samples will be divided into batches of 18, or six samples per batch, identical to those described in Section 5.4.

Measurement of all metals in the leaching solutions will use ICP–MS at TRA. Labware, pH-measurement and control apparatuses, a conductivity probe, and consumables adequate to process leaching studies in batches of 18 (plus the QC load) will be identified or procured. Laboratory space at TRA will be identified.

5.7 Sequential Aqueous Extractions

Actinide speciation will, to a large extent, dictate the ultimate extent of release of the contaminant metal and will provide guidance about remediation options. For example, actinides bound on frayed-edge sites of clays are not apt to solubilize under anything approaching normal conditions; however, carbonate-bound actinides might readily mobilize as a result of a pH incursion. Similarly, reduced actinides would not be expected to mobilize unless highly oxidative conditions developed.

Generating chemically explicit speciation information for the actinides is difficult, which has discouraged widespread attempts to do this. However, starting with Tessier in 1979, the technique of sequential aqueous extractions (SAE) (Ibrahim and Salazar 2000; Litaor and Ibrahim 1996; Loyland et al. 2000; Tessier, Campbell, and Bisson 1979) has made valuable contributions to the understanding of how actinides are chemically distributed among different soil mineral assemblages. Sequential aqueous extractions do not provide explicit chemical speciation, but do divide the subject metal contaminants into operationally defined categories of species that enable the user to effectively project how chemical perturbations to the system will impact the solubility of actinides in the system.

Three 1-g replicates from each of the 42 soil and waste samples will be subjected to SAE to determine the operationally defined speciation of the actinide contamination. This information is needed because the chemical form of the actinides will largely dictate appropriateness of in situ remediation approaches. This information also will enable estimation of actinide release from a secondary source (i.e. contaminated soil) as a result of oxidative or reductive changes resulting from an in situ treatment.

The SAE process was developed by Harris and coworkers at the INEEL and is a modified version of the original Tessier procedure (Tessier, Campbell, and Bisson 1979). It divides operational speciation into five categories and, for this reason, four wet chemical manipulations are needed. Each manipulation is followed by an ICP–MS analysis of the leachate. The five speciation categories are:

1. Cation exchangeable
2. Carbonate bound
3. Organic bound (oxidizable)
4. Fe/Mn sesquioxide bound (reduceable)
5. Residual.

The graphite subsamples also will be subjected to SAE, if sufficient sample is available.

Replicates of the samples will be divided into batches of 18, or six samples per batch, as in Subtask 5.9.5.

Measurement of all metals in the leaching solutions will use ICP–MS at TRA. Labware, pH-measurement and -control apparatuses, a conductivity probe, and consumables adequate to process leaching studies in batches of 18 (plus the QC load) will be identified or procured. Lab space at TRA will be identified.

5.8 Column Studies

Geochemical transport behavior of plutonium and the other actinides is needed to validate transport predictions at OU 7-10 relevant to removal or in situ treatment. Parameters (e.g., as adsorption and release) are influenced by variations in the Eh and pH, and coupling between these processes and advection or film flow will affect overall transport phenomena. Testing will be modified to optimize transport of actinides because this will provide an upper bounding of transport. The Eh and pH modifications will be guided by results of earlier testing. Column studies, in which controlled actinide species and concentrations will be added as the influent, will be conducted.

Columns will be packed with RWMC sediments, and column volumes will be measured using a nonretained tracer such as I⁻. Columns will be loaded with actinide solutions (e.g., concentration to be determined and speciation to be varied). Actinides will be eluted using simulated groundwater having adjustable pH. Actinide breakthrough will be measured in terms of elution volumes. System perturbations, including addition of complexing ligands and exchangeable cations, will be conducted to determine how susceptible the actinides are toward solubilization and transport. When actinides are eluted, the speciation of the actinides will be determined.

Column apparatus located within the α wing at TRA will be identified or procured for this task. ICP-MS capability at TRA will be used for analysis of the eluate solutions.

5.9 Colloids and Organic Complexes

The subtask structure is designed to provide a detailed investigation of the role of colloids and organoactinide complexes as factors that could be responsible for rapid mobilization of a small fraction of the actinide contaminants in the RWSC samples. If the TRU-bearing waste must be excavated and removed, then an understanding will be needed about whether the surrounding soil material constitutes a secondary source capable of releasing a fast-moving actinide fraction. The same information will be needed if waste is treated in situ, because physical and chemical perturbations could result in conditions that favor release of colloid-bound or complexed actinides.

Tasking for the colloids addresses whether or not (1) a significant colloid population could be suspended from the soil and waste, (2) the colloid fraction contains actinides, and (3) the actinide-bearing colloids are mobile. Research into the role of organoactinide complexes has similar objectives to determine whether:

- Organic or bio-organic ligands are present
- Soluble complexes are formed with the actinide contaminants
- Complexes formed are transported.

The tasking emphasizes separation of colloids and complexes from the soil and interstitial samples, and subsequent physical and chemical characterization of the colloids. A mobility assessment and examination of factors responsible for formation of colloids and complexes will be the focus of later testing. The existence of a small, but fast moving, actinide fraction has been widely speculated; however, little information is available that would provide insight into its physical and chemical nature or transport behavior, and effectively nothing specific to the RWMC site. The influence that in situ remediation approaches will exert on the fast-moving fraction must be understood if the risk associated with this fraction is to be assessed.

5.9.1 Subtask 5.9.1—Colloid Separation

The RWSC interstitial soil and waste samples will be subjected to nanopure water extractions, which will have the effect of suspending colloids. If colloid-facilitated transport of actinides is a significant factor, then they should be suspendable under relatively modest conditions (e.g., a low ionic-strength water infiltration).

Three different types of actinide-bearing colloids have the potential to impact actinide transport, and must be considered separately because their composition is substantially different:

- **Pseudocolloids** are defined as (principally clay) particles less than 1 μm in size, are suspendable in low ionic-strength water, and may be highly adsorptive toward contaminants. Actinide-bearing pseudocolloids will form if clay colloids are suspended during a low ionic-strength infiltration and will function to adsorb actinides.
- **Intrinsic colloids** comprise actinide cationic species that will polymerize subsequent to hydrolysis. It is possible that these polymers will be suspended (i.e., are colloidal) and then transported despite the fact that the actinide is apparently exceeding the solubility limit.
- **Bio-organic colloids** are formed from the interaction of molecular microbial detritus with actinide, forming a macromolecular assembly that is suspended and contains actinides. As in the previous two cases, the bio-organic colloids may be readily transported.

This subtask would involve suspending colloids using a very low ionic-strength water leach, followed by removal from solution using a combination of centrifugation and filtration. The fraction of mass accounted for by the colloids will be measured.

5.9.2 Subtask 5.9.2—Colloid Characterization

Understanding the physical and chemical character of the colloids is critical for assessing the likelihood of actinide transport in colloidal form. For example, the presence of actinide-bearing pseudocolloids would implicate the fine clays and water infiltration events as mobilization factors. Observation of intrinsic colloids would indicate the presence of high concentrations of actinides and reducing conditions forming cations capable of hydrolysis. The presence of bio-organic colloids implicates natural organic matter or microbial action. This information would enable assessment of in situ remediation strategies under a variety of conditions.

Analytical characterization will be performed on the separated and unseparated colloids to make this determination. A fraction of the separated colloids will be aggressively dissolved and analyzed using ICP-MS to determine total actinide content. The colloids also will be subjected to surface analysis using SIMS, which will permit colloid categorization, because the SIMS spectra of the three types are vastly different. Finally, unseparated colloid suspensions will be analyzed directly using ESI-MS. For colloids that are in a size range that resists precipitation (greater than 50 nm), the ESI-MS will enable analysis of the unseparated solution. As in the case of SIMS, the ESI-MS fingerprints of the three types of colloids are significantly different and easily distinguished from one another.

5.9.3 Subtask 5.9.3—Separation of Organoactinide Complexes

Possible formation of organoactinide complexes is a potential transport issue because of the presence of organic chelating agents like ethylene diaminetetraacetic acid (EDTA) and diethylenetriaminepentaacetic acid (DTPA) present and disposed of in the waste mixtures at the RWMC.

These organic molecules will occupy coordination sites around the actinide cations in solution, which will interfere with adsorption of the actinides to the active sites on the mineral matrix. If the complexes are stable, they may resist adsorption and remain in solution for a long time and over great distances. The problem is complicated by the presence of microbially derived compounds consisting of multiple chemicals that are functionally related but have dissimilar compositions and structures.

The complexes can be separated from the solid samples by using a modified extraction procedure in which the polarity and or hydrophilicity of the solvent system used is decreased compared with water. This will result in extraction of organics that might be responsible for strongly binding to actinides in solution. A second approach that will be pursued will be to use high performance liquid chromatography.

5.9.4 Subtask 5.9.4—Characterization of Organoactinide Complexes

The principal tool for characterization of the complexes will be mass spectrometry. The ESI-MS is ideally suited for identification of both complexed and noncomplexed actinide species and organic ligands. Understanding the chemical nature of the complexes will enable scientifically defensible correlation with mobility behavior.

The subtask structure is designed to provide an actinide mobility assessment and examination of factors responsible for formation of colloids and complexes. Modeling of actinide mobility will require validation at some level. (Note that previous testing provided only release information.) It is impossible to present realistic surrogates for actinides (especially plutonium) because of the wide range of unique chemistry that they display; however, column transport studies would provide such validation.

5.9.5 Subtask 5.9.5—Colloid Transport in Operable Unit 7-10 Sediments

Little information is available in literature about colloid-facilitated transport of actinides. Quantification of the risk associated with colloid transport will be required because migration of even small fractions of actinide contamination from a secondary source after exhumation, or from pits subjected to in situ remediation schemes, are and will remain a significant concern that will have to be rigorously addressed. Column transport studies will be conducted in which actinide tracers will be adsorbed to pseudo colloids; these will be suspended and used as column influent. A second type of test will be designed to simulate higher plutonium concentrations at the head of a column, which then will be saturated and driven anoxic (reducing), in an attempt to generate reduced plutonium species capable of hydrolysis and polymerization. Subsequent initiation of flow will optimize chances of observing intrinsic plutonium colloids. Bio-organic colloids will be synthetically generated from plutonium solutions and humic and fulvic acids. The resulting colloids will be characterized and used as influent to column transport experiments.

This suite of testing will enable evaluation of the possible role that all three types of colloids might play in actinide transport and will determine whether colloid-facilitated transport is a significant concern for actinide mobility from a secondary source or from a pit treated with an in situ remediation scheme.

5.9.6 Subtask 5.9.6—Transport of Organoactinide Complexes in Operable Unit 7-10 Sediments

A variety of anthropogenic organic complexing agents were used in plutonium operations. An example is EDTA, which forms strong ligand complexes with actinides. Ligands like EDTA will interfere with binding of the cationic-solution-phase actinides to anionic active sites present on the surfaces of the mineral matrix (i.e., OU 7-10 sediments). Assessment of the role of organoactinide complexes in fostering development and transport of a fast-moving fraction will be critical for assessing release from a secondary source subsequent to exhumation, and for assessing release from a grouted or thermally treated actinide-

bearing pit. If testing reveals that organoactinide complexes are present, then this subtask will provide initial transport data needed for expanding modeling to account for the effect of their presence.

To accomplish the testing, organoactinide complexes will be synthetically generated and used in column-transport experiments. Comparison with behavior of uncomplexed actinides (see Section 5.8) will reveal whether the presence of ligand complexes is responsible for accelerated transport.

This testing requires some understanding of the identity, quantity, and actinide-content of the colloids present in the RWSC samples. It will require ICP–MS instrumentation (located at TRA), ESI-MS (to be located at TRA), ion trap-secondary ion mass spectrometry (located at TRA), filtration and centrifugation equipment, and labware and consumables adequate to perform colloid and organoactinide extractions and separations.

6. IMPLEMENTING PROCEDURES

6.1 Management and Technical Procedures

Several INEEL internal procedures will be followed for the RWSC studies, specifically:

- MCP-2875, “Maintaining Laboratory Notebooks”
- MCP-2669, “Hazardous Material Shipping”
- MCP-557, “Managing Records”
- MCP-3571, “Independent Hazard Review”
- MCP-7, “Radiological Work Permit”
- MCP-70, “Mixed Waste”
- MCP-3469, “RCRA Satellite Accumulation Areas”
- IAG-117 and IAG-118, Interface agreements between TRA and Waste Generator Services.

6.2 Sample and Analysis Management

All samples will be managed in accordance with TRA sample management procedures. Because all data to be generated will be experimental, it will be contained in laboratory notebooks and archived according to MCP-2875 and MCP-557.

7. SCHEDULE

The project schedule will depend on funding availability. Table 7 shows the tentatively planned schedule.

Table 7. Tentative schedule for retrieved waste and soil characterization.

Activity	Start	Finish
Samples received at TRA	—	11/7/2003
Total actinides analysis of soil	11/10/2003	1/19/2004
Total actinides analysis of waste	1/22/2004	3/8/2004
Leachability of soil	1/5/2004	2/24/2004
Leachability of waste	2/25/2004	4/7/2004
Sequential extraction of soil	3/25/2004	5/26/2004
Sequential extraction of waste	5/27/2004	7/9/2004
Material characterization	1/20/2004	4/5/2004
Interpret data and report	6/18/2004	9/30/2004
Column studies	10/01/2004	9/30/2005
Colloids and organic complexes	10/01/2004	9/30/2005

TRA = Test Reactor Area

8. QUALITY ASSURANCE

8.1 Data Quality Objectives

The objectives of this test plan are to understand actinides in Pit 9, their concentrations, and the extent of release from the samples provided by the OU 7-10 Glovebox Excavator Method Project. Thus, data quality must support conclusions to be generated from these experiments.

8.1.1 Sample Acquisition

Guidance for the sampling and sample labeling are provided by the FSP.

8.1.2 Sample Shipment to the Test Reactor Area and Return of Excess Material

Sample labeling and manifesting used for shipment from the OU 7-10 Project to INTEC will be maintained.

8.1.3 Visual Inspection and Subsampling at Test Reactor Area

Results of all visual inspections will be recorded in assigned laboratory notebooks. Subsampling will maintain the sample-labeling scheme and will be appended with one alpha or numeric designator to indicate a subsample. A log of all samples and subsamples will be established in an assigned laboratory notebook.

8.1.4 Analysis for Total Actinide Content in Subsamples

Quality control for this task will be based on a modified SW-846 procedure. This will enable analytical precision and accuracy to be defined, which in turn will enable variability between samples to be assessed. Subsampling precision will be evaluated by comparing results from the three duplicate subsamples. If results vary by more than 30% relative percent difference, then additional subsamples will be taken and analyzed. Matrix spikes will be included in the analytical protocol, to assess extraction recovery and analytical accuracy. Duplicate analyses of the same sample will be performed periodically to assess analytical precision. Instrument blanks will be performed periodically, and especially after many positive analyses. Inductively coupled mass spectrometry calibrations will be performed daily when samples are analyzed. Results of all QC operations will be recorded in laboratory notebooks.

Glassware cross contamination is a potential issue for samples that have substantial actinide concentrations. For samples that have higher concentrations, glassware blanks will be performed, and if these are positive, glassware will be discarded to eliminate the possibility of cross contamination.

8.1.5 Characterization of Sample Mineralogy, Surface Chemistry, and Selected Chemical and Physical Properties

A trained X-ray diffraction technician will conduct X-ray diffraction identification. Secondary ion mass spectrometry spectra will be acquired subsequent to daily mass calibration and sensitivity check. A trained technician will conduct surface area analysis, and accuracy will be validated by analysis of standards.

8.1.6 Contaminant Leachability Across pH- and Ionic-Strength Gradients

For the leaching studies across a pH gradient and the sequential aqueous extractions, only the data quality objectives focused on analytical performance are applicable because there is no standard actinide-bearing soil that could be a reasonable mimic for the plutonium-bearing material anticipated from OU 7-10. This reasoning also holds for the column studies and the colloid and complexes task. Analytical QC will follow that described in Section 8.1.4.

8.1.7 Sequential Aqueous Extractions

See Section 8.1.6.

8.1.8 Column Studies

See Section 8.1.6.

8.1.9 Colloids and Organic Complexes

See Section 8.1.6.

8.2 Test Plan Modification

Revisions to this document will follow the requirements set forth in MCP-233, “Process for Developing, Releasing, and Distributing ER Documents (Supplemental to MCP-135 and MCP-9395).” Therefore, with the exception of field changes (see paragraph below), changes must be submitted on a “Document Action Request Form” (Form 412.11) and reviewed and approved by the same disciplines and stakeholders that approved the original document. This ensures that necessary changes can be made and that all stakeholders have the opportunity to provide comments on the suggested changes before they are implemented.

Management Control Procedure-233 defers to MCP-135, “Creating, Modifying, and Canceling Procedures and Other DMCS-Controlled Documents,” for field changes. This MCP specifies requirements for field changes. A field change is defined as a necessary change to a technical procedure in response to an unanticipated procedure or error to facilitate continuation of test operations that might otherwise stop or be unreasonably delayed. The principal investigator and project manager will review any proposed field changes. Changes will be reviewed for technical accuracy and compliance with environmental, safety, and operations requirements and to ensure that the test plan is still usable. The project manager is responsible for obtaining the appropriate reviews and approvals as specified below and for documenting the action on a “Document Action Request Form.” A redlined copy of the approved changes will be distributed to the project team and Document Control.

Note: Management Control Procedure-135 allows no more than five field changes.

9. HEALTH AND SAFETY

Health and safety associated with the RWSC studies will be managed using the independent hazard review process (MCP-3571), which will be conducted for all aspects of this test plan. In addition, all activities that handle radioactive material will be subject to generation and approval of a radiological work permit (MCP-7).

10. WASTE MANAGEMENT

Investigation-derived waste will be generated during the testing. Disposal of waste generated during testing will be handled according to the guidelines established in the *Waste Management Plan for the OU 7-10 Glovebox Excavator Method Project* (INEEL 2003) and the guidelines at the facility where the work is conducted. All workers will practice current waste-minimization strategies. Anticipated waste streams are discussed below.

10.1 Testing Waste

Waste generated from bench analyses will include:

- Laboratory debris
- Leachates and modified OU 7-10 soil and waste.

The largest volume will be the leachates and modified OU 7-10 soil, which will be recombined. Based on analysis conducted during the project, explicit knowledge of the actinide contents will be available, which will enable the material to be handled as mixed waste (MCP-70).

10.2 Waste Minimization and Pollution Prevention

The tests are designed around waste-minimization and as-low-as-reasonably achievable concepts. The smallest amount of radiologically spiked and hazardous matrix samples needed for valid analytical data will be prepared. Activity of the SDA samples will be the primary factor in determining as-low-as-reasonably achievable and waste-minimization strategies for these samples. Approved handling in all stages of testing will minimize waste and radiological contact.

10.3 Handling, Storage, and Disposition

All routine waste generated in sample preparation and analysis will be disposed of by the analytical facility(ies) in accordance with individual bench-disposal practices, such as those outlined in MCP-3480, “Environmental Instructions for Facilities, Processes, Materials, and Equipment.”

11. DATA ANALYSIS AND INTERPRETATION

11.1 Data Evaluation and Analysis

All quantitative data will be checked to assess data quality and to quantify accuracy of this simulation and validity of the tests and analytical technique. This data check will be made using two parameters: precision and accuracy.

All values have a statistical uncertainty due to heterogeneity of the sample and instrument variations. The samples will necessarily be highly heterogeneous; therefore, evaluation of representativeness is outside the scope of this project. However, instrumental error can be quantified by evaluating spikes, blanks, and replicates.

Quality assurance and QC samples are routinely prepared to verify analytical precision and accuracy including spikes, blanks, replicates, and standards. Precision of the test measurements, types of data that will be collected for each test activity, and data quality expected in the measurements are outlined in the following subsections. Relative percent difference of duplicate samples is used to assess precision. Accuracy is measured using standards and percent recovery of matrix spikes. The difference in replicate samples determines the minimal difference between nonreplicate samples considered to be significant.

All data will be checked for accuracy and precision initially by an instrument or an operator and finally by the principal investigator. A second investigator will review data on a daily basis for objective, accuracy, and completeness. The principal investigator will review the procedures and data at least weekly for concurrence with the test plan. If the quality assurance objectives are not met, then an alternative approach will be identified to better quantify accuracy and precision.

11.1.1 Spikes

A spike is the addition of known amounts of the analyte (e.g., plutonium) to a known volume of sample to test for any matrix affects. This is specifically applicable to the total actinide content determinations, but is not in any way applicable to either the studies of leaching across a pH gradient, the sequential aqueous extractions, the column studies, or the colloids and complexes task.

For the total actinide content experiments, each spike a percent-recovery (%R) is calculated using Equation (1):

$$\%R = [(SSR - SR) / SA] \times (100) \quad (1)$$

where

SSR = Spiked sample result

SR = Sample result

SA = Spike added.

Percent-recoveries between 75 and 125 indicate that the matrix is not interfering by enhancing or detracting from the determined concentrations of analytes. Closeness of bench-duplicate and spike results indicates the precision and matrix effects associated with the bench operations but not sample collection.

11.2 Document Management and Sample Handling

The sample and surrogate preparation activities and test results are documented in an assigned, bound notebook, which will serve as the primary record for subsequent data reduction.

11.2.1 Data

All bench data and observations will be recorded in a bound, library-registered, laboratory notebook. It will be dated and signed daily by the person doing experiments. Entries will be signed weekly by a second person. All reagent labels are recorded completely in the notebook

Analytical data will be compiled on spreadsheets. Data for each sample will vary depending on the experiment performed. However the finalized procedure, and requisite data for each experiment will be identified by a meeting between the scientific staff before initiation of the analyses.

All log books, and other forms of raw data will remain in the custody of INEEL and the principal investigator for review until the release of the logbooks for long-term storage. All the reduced data will also be store in the project file. The published INEEL reports remain on file and are available through the library system.

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